# THE ANALYST.

JULY, 1892.

## PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

The usual Monthly Meeting of the Society was held on Wednesday, 1st June, the President (Mr. Hehner) occupying the chair.

The minutes of the last meeting were read and confirmed.

The following gentlemen were proposed as members:—A. P. Aitken, D.Sc., Chemist to the Highland and Agricultural Society of Scotland, 8, Clyde Street, Edinburgh; Frederick Wm. Tompson, F.C.S., Chemist in the employ of Messrs. Bass & Co., Brewers, Burton-on-Trent, Stapenhill Road, Burton-on-Trent; and John Williams, Private Assistant to Professor J. B. Harrison, Government Analytical Chemist, British Guiana, Georgetown, Demerara, was proposed as an associate.

Messrs. Samuel Francis Burford, F.C.S.; James William Westmoreland, F.I.C., were duly elected members of the Society; and Messrs. G. H. Gabb and W. N. Yarrow, were duly elected associates.

Mr. H. Droop Richmond read the following "Notes from the Khedivial Laboratory, Cairo":—(7) "On a Sample of Milk yielded by an Arab Woman." (8) "Egyptian Flour." (9) "Some Egyptian Waters." (10) "The Manganese beds of Assiout." The publication of these papers is unavoidably held over until next month.

In the absence of Dr. Leather, Mr. Dyer read the following paper:—

### THE DETECTION OF CASTOR OIL SEED AND OF CROTON SEED IN FEEDING STUFFS.

By J. Walter Leather, Ph.D., F.I.C.

As is well known, both castor oil seed (*Ricinus communis*) and the seeds of croton (*Croton tiglium*) are very poisonous.

A small admixture, even considerably less than one per cent., of either of these seeds, when present in a cattle food, is sufficient to cause harm and even death. An ordinary

search with the microscope for any seed existing in smaller proportion than one per cent. would, in any case, be not only very tedious, but might fail to detect the constituent.

The difficulties attending a microscopical search for either of these seeds are, however, considerably increased on account of the "seed case" of each being quite impervious to light, and the structure being, in consequence, indiscernable.

The method which I have employed for some time past for the microscopical examination of foods, so far as the recognition of the testa of seeds is concerned, is, firstly, to digest a portion of the material with dilute HCl or $H_2SO_4$ (1 to 2 per cent.) for half-an-hour on the water-bath; then to remove the acid liquid by decantation and to wash the material several times in the same manner; secondly, to digest it with equally dilute KHO or NaHO and to repeat the washing by decantation. In this way I have found that the testæ of all those seeds which one generally meets with in cattle foods, and also the impurities which frequently accompany them, are rendered sufficiently transparent to be readily recognized under the microscope. After washing the alkaline liquid away, it is sometimes an advantage to wash the material with dilute HCl, which reduces the strong colour produced by the action of the alkali. I do not lay any claim to this process, for I believe that many chemists employ both acid and alkali for the above purpose, but I might mention that I have not met in text-books with any directions for their use in the manner I have indicated.

Now, if either castor oil seed or croton seed be subjected to this process, the outer integument of each is washed away, and the principal part of the testa remains black and opaque. At the edges some indication of the structure can frequently be seen.

In any case, owing, as I have said, to the fact that it requires but a very small quantity of either of these seeds, probably 3 or 4 seeds, or less, per lb., to cause serious illness or death to stock, the examination with the microscope of one or two grammes of a material could not be expected to expose their presence with certainty.

During a search, some months ago, for these seeds in some decorticated cotton cake, I subjected a number of pieces which had been picked out with the aid of a pocket lens, to the action of a solution of sodium hypochlorite. I had previously employed this agent to bleach similar materials for another purpose, and I had always found that it removed the colour entirely after a few hours. I therefore hoped that I should in this way obtain a view of the structure of castor oil seed and of the pieces above referred to. To my surprise, however, they remained perfectly unbleached, even at the end of several days. I then subjected a number of pieces of the testæ of both castor oil seed and croton seed (1) to the action of dilute acid and alkali and (2) to sodium hypochlorite. The result was that the seeds remained entirely unbleached. At the same time I treated in the same manner the testæ of cotton seed, *Bassia*, *Polygonum convolvulus*, and locust-bean, all of which, especially the first-named, are very dark-coloured. In every case they bleached quite readily. Free chlorine has a more energetic action, and will gradually bleach both

castor oil seed and croton seed; the time required amounted, however, in my experiments to some three or four days, and even then the colour was not completely discharged. I need not mention other seeds, which are likewise dark-coloured, and which frequently occur in cattle foods, for they present no difficulty in their recognition. I had now in my hands a method by which either castor oil seed or croton seed might be readily separated from other materials, even when present in quite small quantity. If the suspected feeding stuff be first digested with acid and alkali, and be then subjected for a few hours to the action of bleaching powder, any castor oil seed or croton seed would remain black amongst the bleached mass. I may mention here that preliminary experiments had shown me that this agent acted exactly in the same manner as sodium hypochlorite. In order to put the method to a test, I chose undecorticated cotton cake as the material with which to mix the castor oil seed or croton seed. I made this selection because the testa of the cotton seed is just as black after treatment with acid and alkali, as that of either castor or croton.

To one pound of the coarsely ground cotton cake I added, in each of the following experiments, the weighed and coarsely broken up testa of *one seed* of castor and croton respectively, and, after mixing roughly, the whole was digested in dilute HCl and boiled for one hour by passing a current of steam through it. After washing by decantation, the mass was boiled with dilute alkali for a similar period, and again washed by decantation until the washing water was colourless. Bleaching powder, about $\frac{1}{2}$ lb. to 1 lb. (the exact quantity does not appear to be material) was then added, and sufficient water to keep the temperature from rising. This was usually three or four litres. The mass was stirred occasionally and left over night, although the bleaching action was frequently complete at the end of four hours. Washing by decantation now followed, by which means most of the lime could be removed. It remained now merely to pick over, by hand, the bleached material and select the unbleached pieces for examination. This operation requires, for a pound of material, some considerable time, varying from one half to a whole day, but it is so simple that any careful laboratory servant can perform it. Care must be taken not to have anything thrown away. The picking over of the material is most readily performed by spreading small quantities on the bottom of a flat porcelain dish, which is about half full of water; any dark-coloured or black pieces can then be easily seen, and can be taken out. After the unbleached pieces have been separated they are readily examined, and anything other than castor or croton again separated. I may mention here that in a pound of bleaching powder a number of bits of coal or stone may often be found, and, further, in the case of the cotton seed, the hilum does not always bleach so readily as the other portions, so that these would likewise be separated with any castor or croton seed.

EXPERIMENTS—

(1.) Weight of testa of *castor oil seed*, 0·223 grm., mixed with 1lb. of cotton cake: weight of separated castor oil seed testa, 0·200 grm.

(2.) *Castor oil seed testa*, 0·122 grm., mixed with 1lb. of cotton cake: recovered ·087 grm. castor oil seed testa.

(3.) *Castor oil seed testa*, 0·065 grm., mixed with 1lb. of cotton cake: recovered 0·042 grm.

(4.) *Croton seed testa*, weight 0·074 grm., mixed with 1lb. of cotton cake: recovered 0 060 grm.

(5.) *Croton seed testa*, weight 0·080 grm: recovered 0·078 grm.

The method, accordingly, is not merely a qualitative one, but is, at the same time, a valuable quantitative one.

Only in one experiment did I fail to recover the greater part of the seed *testa* which was put into the cotton seed cake. In this case 0·071 grm. of the testa of croton seed was added, and only 0·02 grm. was recovered. An explanation is, however, easily found for this loss. In a preliminary bleaching experiment with castor oil bean, part of a *mouldy* seed was subjected to the action of sodium hypochlorite, and about one-half of the piece became bleached after the lapse of a day. A microscopical examination revealed, however, the fact that this seed-case had been penetrated through and through with a fungus.

In the experiment in question, the croton seed happened to be a mouldy one, and no doubt a part of it became bleached. With regard to the amount of the seeds which were not recovered in the other quantitative experiments, I believe that the loss was due to very minute pieces of the testæ being broken off during the process of boiling, the testa in each case being extremely brittle, and these minute fragments might naturally be overlooked. Usually, I found that the testa of the seed had been broken into some 25 or 30 pieces, by rough pounding with a pestle.

Regarding the hilum of the cotton seed, referred to before as not being completely bleached, there is no difficulty in distinguishing it, for the pieces are round and have no sharp edges; moreover, they are marked by a series of rings alternating grey and black on the surface.

It now remains for me to speak of the peculiarities of the testæ of these two poisonous seeds. The outer covering of each is quite distinct, and, in the case of castor seed, very peculiar. If any pieces of either the one or the other seed be found intact no difficulty will be experienced in recognising them. The outer integuments are, however, extremely thin and brittle, and it must not be expected that they will necessarily be present. Of the two, I have as yet only found castor oil seed in any sample of feeding material, and I therefore cannot give any experience as to the other. In the case of castor oil seed, it may very readily happen that this characteristic integument has disappeared, and as a matter of fact I have often found it to be so.

But in the event of no pieces of either of these seeds being found intact, and if they should have been separated by the bleaching process, they are simply black and opaque and extremely similar. One of their chief characteristics is their extraordinary brittle-

ness, and if one endeavours to cut the testa across with the knife, the two parts fly away some considerable distance, and the broken pieces of either have invariably sharp and frequently ragged edges.

Regarding their thickness, I have measured a number of pieces, and find that of the castor oil seed testa to be from 0·25 m.m. to 0·30 m.m., and that of the croton seed testa to be from 0·20 m.m. to 0·25 m.m.; the latter being thus distinctly thinner than the former.

After passing through the bleaching process, the castor oil seed testa has a much smoother and more shiny exterior than the croton seed, and herein lies one point of distinction.

I have examined the microscopical appearance of sections taken transversely through the testa of each. For the greater part, the structure of each is very similar, each being built up of closely packed bundles of fibres of the same thickness. One difference I have noticed is, that on the outermost edge of the section of the croton testa there is a distinct thickening of each bundle of fibres, whereas this is quite absent from the section of the castor oil seed testa.

Lastly, if the edges of the pieces be examined with a low power objective (about 1in.) with reflected light, the peculiar curvature of the bundles of fibres on the inner edge is readily seen. This is common to both of the seeds.

### Discussion.

Mr. Bernard Dyer said that castor seed found in feeding stuffs was sometimes in the decorticated form. One small piece of husk might then represent a large amount of castor seed.

The President asked Mr. Dyer if this impurity was accidental, or was the seed added intentionally?

Mr. Dyer replied that he was of opinion that sometimes it was added accidentally and sometimes intentionally. In a good many cases it had been found in Brazilian cotton meal and cake; and in that case he believed the castor oil seed had been accidentally introduced, through the castor oil plant growing as a weed among the cotton; but in some cases he was sure that it was deliberately added through ignorance. There was an enormous quantity of castor cake produced, especially in tropical countries, which ought all to be used as manure. It was sometimes bought up by people who were ignorant of its poisonous properties. He had recently had a sample of Niger seed cake sent to him for the purpose of examination, with the view of ascertaining whether it contained castor; and he found that it did. Since then he had found a little castor seed in a sample of linseed cake. He also found a considerable quantity of Niger cake in it, with which the linseed cake had been adulterated, and it seemed not at all improbable that the Niger cake had contained the castor.

Mr. F. H. Perry Coste pointed out with regard to the question that Dr. Leather had referred to at the beginning of his paper—the difficulty of rendering some of those objects transparent—that some microscopical journals had recently given an account of a new process, just discovered, for effecting that purpose. The process was to place the leaf or other object in a drop of glycerin on a slide, cover with a cover-glass, and gently heat, replacing the glycerin from time to time. That method rendered a thick object so transparent that the whole structure of it could be seen. It was, of course, quite different in character from the bleaching process, the colour not being entirely removed. The secret of the process is "the use of glycerin and the saturation of the cells."*

Mr. Bertram Blount wished to ask Mr. Dyer if he could inform him whether any attempt had been made to detect castor oil seed by chemical means? He supposed both it and croton seed would be susceptible of detection by recognising the poisonous principles they contained. He believed it was a fact that a poisonous principle had been definitely recognised in castor. As Mr. Dyer had said, there might be only a small quantity of the husk to a comparatively large quantity of the seed, and thus any method of detection, based on the recognition of the husk, was necessarily imperfect.

Mr. Dyer said that he did not think that the definite poisonous principle of castor had been isolated. Professor Tuson, years ago, isolated an alkaloid, which was described in Watt's *Dictionary of Chemistry*, but if he (Mr. Dyer) recollected rightly, that alkaloid did not appear to have been the active ingredient of the seed.

Sir Charles Cameron said that he had some experience in the examination of oil cakes, and on one or two occasions he detected what he believed to be the impurity referred to by Mr. Dyer. He would say, however, that he did not feel great confidence in the results he had arrived at.

Mr. Cassal asked whether it had been attempted to make use of chlorate of potash and hydrochloric acid in the bleaching process described? Mr. Dyer had stated that it took a very considerable time to carry out the bleaching with hypochlorite. He (Mr. Cassal) had found the chlorate of potash and hydrochloric acid method very rapid for vegetable tissue generally.

Sir Charles Cameron said that on one occasion he extracted, by purely chemical treatment, a substance such as that referred to from croton seed, which had strong blistering properties. Its ether extract had acted as a vesicant upon his wrist. It was believed that the cake, in that case, had been very injurious to animals.

Mr. Dyer, replying to Mr. Cassal, said he had not tried the chlorate of potash and hydrochloric acid method. He would expect that method to bleach almost anything. The process Dr. Leather had described was not at all tedious. It was necessary to get rid of the mealy, farinaceous or albuminous part of the meal. That was easily done in an hour or so by the alkali and acid treatment. If the bleaching were

* See "Microscope," xi. (1891), pp. 265-7; and Royal Microscopic Society's Journal, 1892, pp. 287-8.

set going at night, the sample was always ready the next morning. He believed it would be advantageous to use a solution of sodium hypochlorite, which would be free from many of the impurities which were indicated in the paper likely to be present in ordinary bleaching powder. Dr. Leather found that sodium hypochlorite would act in every respect as well as bleaching powder. He thought that the point mentioned by Dr. Leather, with reference to the hilum of the cotton seed, should be carefully noted. The part of the cotton-seed husk which included the hilum did not bleach as a rule, and at first sight looked a little like "unskinned" castor husk; but it was always soft and fleshy at the edges, and if looked at with a low-power magnifying glass, bore no resemblance to castor husk. The latter, when magnified, looked very like a much-diminished fragment of smooth cocoa-nut shell.

Sir Charles Cameron asked Mr. Dyer what he had found certain small particles in decorticated cotton-cake to consist of? The particles in question were very hard and brittle.

Mr. Dyer replied that, if the particles occurred in Brazilian cotton-cake, they were most likely castor.

This terminated the proceedings of the Society.

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## ESTIMATION OF FAT IN MILK BY THE BABCOCK METHOD.

By Alfred W. Stokes.

*Read at Meeting, May 4th*, 1892.

In the year 1889, Dr. Babcock, of the United States, introduced a method of determining fat in milk, which could be used by dairymen, and which was rather largely used by the dairymen of the United States and Canada.

This method is now being, to some extent, taken up in England.

About a year-and-a-half ago, Mr. G. H. Claflin, of the Franklin County Creamery, Vermont, kindly gave me his experiences of the method, and spoke very highly of it as a dairy process.

Having lately had an opportunity, through the kindness of Messrs. Welford and Sons, of testing the apparatus, I lay my conclusions before this Society. The essential features are:—A special bottle, one-and-a-quarter inches diameter, and 3 inches high, having a neck, 4 inches long, and $\frac{3}{16}$ inch diameter. This neck is divided into ten parts, these being subdivided into fifths. The whole bottle holds 50 c.c., of which the neck contains about 2 c.c. Into the bottle 17·6 c.c. of the milk is pipetted, and then an equal bulk of strong $H_2SO_4$ is added. The two liquids are mixed together by gentle shaking. Great heat is developed, the mixture turning quite black. While still hot, the bottle is put into a whirling apparatus, in which four or more bottles can be whirled round at the same time. This centrifugal apparatus is very simple, consisting of a large driving wheel,

15 inches diameter, on a horizontal axis, and having a wide, flat side rim, against which a small india-rubber-tired wheel presses tangentially. This latter wheel is part of the vertical spindle that bears the four arms, on which are cups to receive the bottles. The big wheel has a handle, by which it may be turned, one revolution giving $6\frac{1}{2}$ revolutions of the little wheel. There is no gearing, friction being all that is necessary. Though looking slight and inefficient, it works very freely and well.

Having placed the bottle, or bottles, in the whirling apparatus, they are kept revolving for eight minutes, at the rate of from 400 to 500 revolutions per minute. Then the bottles are filled up to the beginning of the neck with hot water, and again whirled, this time for two minutes. More hot water is added to near the top of the neck. After whirling for two minutes longer, the fat, which has risen to the top of the water, is read off. The divisions equal percentages and fifths of percentages of fat. The reading off of the percentage of fat is best done while the bottles are immersed in hot water. Otherwise, the fat is spread over the tube as the cooling mass contracts. The great difficulty is to get an accurate reading of the fat. The fat in the tube is not the usual light yellow butter-fat, but a thick viscid brownish mass, often quite black. If the bottles are left for any length of time, after the whirling is finished, black flocks of altered casein will rise and mix with the fat, making the reading almost impossible. Occasionally this happens, however rapidly the reading is taken. The fat being more viscid than melted butter, rarely forms a good column, with a well-rounded base, and a sharp upper meniscus; so that it is not easy to be sure of the reading to a tenth of a per cent.

In the following list I place some results, obtained on milk samples, in which the fat was very carefully calculated, and frequently also determined by the Schmid or paper-coil process, as compared with the Babcock results:—

| Sample. | Calculated. | Babcock. |
|---|---|---|
| No. 1. | 4·30 | 3·80 & 3·78 |
| 2. | 4·11 | 3·80 |
| 3. | 4·09 | 3·80 |
| 4. | 3·85 | 3·80 |
| 5. | 3·21 | 3·00 & 3·10 |
| 6. | 3·17 | 3·00 & 3·00 |
| 7. | 3·12 | 2·80 |
| 8. | 3·05 | 3·00 |
| 9. | 3·00 | 3·00 |
| 10. | 2·86 | 2·80 |
| 11. | 2·26 | 2.10 & 2·00 |
| 12. | 1·70 | 1·20 |

Here we see differences ranging from 0 to 0·5 per cent., with an average difference of 0·21. It is possible that with longer and more experienced working with the apparatus,

the differences might be somewhat less. Never does the Babcock method give results higher than those by calculation.

In an article on this method by Mr. F. T. Shutt (*Chemical News*, vol. 64, No. 1,649), he likewise does not find it to give results above those of reliable gravimetric processes. His average difference he states to be between one and two-tenths of a per cent.

By the kindness of Mr. J. Hampton, the manager of "The Devizes Dairy and Farmers' Supply Co.," I am enabled to place before you a list of fat determinations, on the same samples, by Mr. Hampton, using the Babcock method, and by myself, using the calculation method. As you will see by this list (not printed here), of 18 milks, the results given by the Babcock method are from ·08 to ·44 per cent. lower than the calculated results, with an average difference of ·19. In no case here does the Babcock process show results as high even as the calculated fat.

Looking at one of the bottles after the process is finished, one notices at the bottom a small white sediment; above this is a black liquid, in which floats a quantity of black caseous material; above this, in the neck, is a quantity of water, surmounted by a brown or black viscid column of fat. Examining, microscopically, the white sediment, it is found to consist of a mass of crystals, mixed with a larger quantity of fat globules. It is clear that all the fat does not rise through the dense mixture of acid and milk. More or less fat, also, is distributed through the mass of black liquid. When the results do agree with the actual fat present, it is either due to an allowance made in calibrating the neck, or else to some casein being taken up by the floating fat that is measured at the top of the tube.

The white crystalline sediment I find to be principally sulphate of lime. The process is in principle the same as the Werner-Schmid process, which came out the previous year. Only, instead of artificially boiling the milk with hydrochloric acid, advantage is taken of the heat, naturally produced, on mixing milk with sulphuric acid. And then, instead of dissolving out and weighing the fat then set free, it is measured, after being driven together by a centrifugal apparatus. When working at the Schmid process in 1888, I tried the effect of using sulphuric acid, instead of hydrochloric acid; with benzoline afterwards to extract the fat, ether not being admissible. I found that after mixing the milk and acid, the fat is not all at once set free; for if the mixture be cooled five minutes after mixing, then another sample be cooled ten minutes after mixing, and a third sample be left to cool naturally, most fat is given by the third sample, least by the first sample.

Leaving the mixture to cool, I did not get all the fat actually present in the milk; hence, I then concluded that sulphuric acid destroyed some fat. Though not a good laboratory process, the Babcock method is, in my opinion, the best, cheapest, simplest, and most rapid dairy method of determining fat in milk. The use of strong, almost boiling, sulphuric acid is, however, somewhat dangerous.

In my hands, the process altogether failed for sweetened condensed milks. For much of the work, and for the diagrams shown to-night, I am indebted to my assistant, Mr. W. N. Yarrow.

Discussion.

Mr. H. Droop Richmond thought the method described showed up very badly when compared with the Leffmann and Beam method. It seemed to have a considerable number of drawbacks in manipulation, which were not shared by the latter. But that was a comparatively unimportant point, when it was considered that the results obtained with the Babcock process by Mr. Stokes were inaccurate, while those quoted by the President, by the Leffmann and Beam method, were substantially exact. He was inclined to give every preference to the latter method.

Mr. Woosnam stated that he had recently made some experiments with the Babcock process, but the machine he used had niches for 30 bottles instead of 4. The results were unsatisfactory, and very similar to those obtained by Mr. Stokes. He did not think that sulphuric acid was a fit substance to be put into the hands of dairymen or others unskilled in manipulation.

The President thought that the Adams process was very difficult indeed to beat; it was the standard by which other methods were to be judged. But if a method entailing less trouble, and by which quicker determinations could be made, were brought out, he had no doubt that it would be welcomed by public analysts.

Mr. Stokes explained that instead of heating the tube artificially, it was heated by the mixture of sulphuric acid with milk.

Mr. Sidney Harvey thought that as the Babcock machine had no cogwheels it was the better of the two.

The President stated that Messrs. Leffmann and Beam only claimed for their process the improved method of liberating the fat.

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**Resume of a Research on the Acids of Butter. Emil Koefoed.** (*Bulletin de l'Academie Royale Danoise*, 1891.) 785 grammes of a butter fat giving 15·1 c.c., Reichert figure (by Nilson's modification *Z. f. Anal. Chem.* 28, 175), were saponified with 200 grammes caustic soda in 500 c.c. of water; the soap was decomposed by 300 grammes of sulphuric acid diluted with 500 c.c. of water, and the liquid boiled under an inverted condenser till the soap was all decomposed, a current of carbon dioxide being passed through the flask. The fatty acids (720 grammes) were filtered, and the aqueous filtrate shaken three times with ether, which, on distillation, left 6 grammes of acids smelling like butyric. This extraction with ether was proved to have removed the whole of the organic acids.

The 6 grammes constituted Portion 1.

The 720 grammes of acids left on the filter were distilled under 30 mm. pressure. The acids distilling between 93° and 200° weighed 54 grammes, and constituted Portion II.

The remaining acids were dissolved in 500 grammes of alcohol of 95° Tralles, and were several times crystallized from this medium ; 100 grammes were thus obtained.

The whole of the alcoholic filtrates (about 4 litres) were, after the addition of 30 grs. of acetic acid, treated with an alcoholic solution of 600 grammes of crystallized lead acetate. The precipitate was collected after 24 hours on a filter, washed with alcohol, and air dried. The acids were then set at liberty by boiling with hydrochloric acid, and weighed 314 grammes, which, with the 100 grammes obtained by crystallization, formed Portion III.

The filtrate was made faintly alkaline with ammonia, and a small quantity of a semi-fluid lead salt separated, probably Gottlieb's oxyoleate of lead. This was boiled with hydrochloric acid, and the acid thus obtained dissolved in ammonia, and its barium salt precipitated by barium chloride. This formed Portion IV.

From the filtrate the alcohol was removed by distillation, and the fatty acids transformed into barium salts as above. These constituted Portion V.

The Portions were then examined.

*Portion V.* The barium salts were boiled successively with acetic ether and chloroform. From the acetic ether solution, oleate of barium separated on cooling. This was re-crystallized from 80 per cent. alcohol, and then gave the following figures :—

| | Found | Calculated for barium oleate. |
|---|---|---|
| Ba ... ... | 19·38% ... ... | ... 19·59 |

The chloroform solution gave, after cooling and filtering, on the addition of four volumes of ether, a white amorphous precipitate, which, on drying, became a brown, gummy amorphous mass.

The figures of analysis were as follows :—

| | Found | Calc. for Ba. $(C_{15} H_{27} O_4)_2$ |
|---|---|---|
| C ... ... | 52·99 ... ... | ... 54.80 |
| H ... ... | 7·51 ... ... | ... 7·98 |
| Ba ... ... | 20·07 ... ... | .. 20·24 |

The author regards this acid as $C_{15}H_{28}O_4$. It easily decomposed, as after some time its barium salt becomes insoluble in chloroform.

*Portion IV.* This is insoluble in ether, acetic ether and chloroform. The analysis is :—

C 56·43 ... H 8·43 ... O 12·69 ... Ba 22·45

The author assigns the following formula to this acid :—

$C_{29} H_{54} O_5$ (dibasic).*

* Probably a mixture.—*Abs.*

*Portion III.* This portion was fractionated under 30 mm. pressure; Bruhl's receiver (*Ber.* 1888, 3339) being employed.

Fractions were collected:—

| | | | |
|---|---|---|---|
| A | 200° | — | 230° |
| B | 231° | — | 238° |
| C | 240° | — | 242° |
| D | 244° | — | 248° |
| E | 251° | — | 255° |

The two last fractions were crystallized from a small quantity of alcohol.

The fractions B—E were fractionally precipitated by quantities of 25 c.c. of a normal magnesium acetate solution. The fractions were then boiled with hydrochloric acid, and the melting points of the acids taken; they were then transformed into silver salts, and the silver estimated. Fraction $E_i$ melted at 68°; after re-fractionating the melting point rose to 69°, and the silver salt contained 27·57 per cent. Ag. (calc. for stearic acid 27·62 per cent.) $E_{iii}$ melted at 62°, and its silver salt gave 29·73 per cent. Ag. (calc. for palmitic acid 29·75 per cent).

*Fraction E* was then principally palmitic acid with a small quantity of stearic acid. Arachidic acid was not detected.

*Fraction D* was also palmitic acid.

*Fraction C* (weighing 131 grammes) was dissolved in 700 grammes of alcohol, and deposited, after 24 hours, 31·5 grammes of solid acids, principally palmitic acid; the remainder was fractionally precipitated by magnesium acetate, seven fractions being obtained.

*Fractions* $C_{iii}$—$C_{vii}$ were magnesium myristate; the acid fused at 53°, and the silver salt contained 32·22 per cent. Ag. (calculated for myristic acid 32·24 per cent.).

*Fraction B* still contained myristic acid; $B_{iv}$ and $B_v$ were, however, lauric acid, for the acid melted at 43·5°, and the silver salt contained 35·35 per cent. Ag. (calculated for lauric acid (35·18 per cent.).

*Fraction A* was neutralized with ammonia, and fractionally precipitated by 5 portions of 20 c.c. of normal alcoholic silver nitrate. $A_{iii}$—$A_v$ corresponded to silver caproate.

*Portion II.* This was fractionated as fraction III A by means of 10 portions of silver nitrate. These were washed with alcohol, boiling water, and again alcohol, and air dried.

*Fraction* 4 contained a percentage of silver corresponding nearly to silver caprylate; it was, therefore, refractionated, and a series of precipitates were obtained corresponding exactly to silver caprylate.

*Fractions* 5—10 were chiefly silver caproate. In order to decide whether this was normal caproic acid or isobutylacetic, the author determined the solubility of the calcium salt. 100 c.c. of water at 17·5 dissolved 2·58 grammes of anhydrous calcium salt

Lieben (*Ann.* 165, 118) has shown that at 18·5° 100 c.c. of water dissolves 2·707 grammes of normal calcium caproate and 11·3 grammes of calcium isobutylacetate.

Butter, therefore, contains normal caproic acid.

*Portion I.* This was principally butyric acid. The silver salt, after crystallization from water, gave 55·27 per cent. Ag. (calculated for butyric acid, 55·38 per cent.)

Grünzweig (*Ann.* 162, 215) has already shown that the butyric acid in butter is normal.

The butter then examined contained 91·5 per cent. of fatty acids, of which the percentage composition is as follows :—

| | |
|---|---|
| Oleic Acid ... ... ... | |
| Acid of the formula $C_{15}H_{28}O_4$ | 34·0 |
| „ „ „ $C_{29}H_{54}O_5$(?) | |
| Stearic Acid ... ... ... ... | 2·0 |
| Palmitic Acid ... ... ... ... | 28.0 |
| Myristic Acid ... ... ... ... | 22·0 |
| Lauric Acid ... ... ... ... | 8·0 |
| Capric Acid ... ... ... ... | 2·0 |
| Caprylic Acid ... ... ... ... | 0·5 |
| Caproic Acid ... ... ... ... | 2·0 |
| Butyric Acid ... ... ... ... | 1·5 |
| | 100·0 |

H. D. R.

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**A New and Delicate Indicator for Alcoholic Solutions. S. Ruhemann.** (*Jour. Chem. Soc.*, lxi. 286).—The author suggests the use of the imide of dicinnamylphenylazimide as a delicate indicator with alcoholic solutions; the change is from red (acid) to purple (alkaline), and the colour is intensified by heating; it can be used to determine the small amount of alkali dissolved from glass by alcohol. The blue (purple) alkali salts are decomposed gradually by water, the ammonium salt being especially unstable. The indicator is best prepared by adding the calculated quantity of bromine (2 mols), to ethyl cinnamate dissolved in chloroform; the solvent is evaporated, and the resulting mass quickly heated with twice its weight of phenylhydrazine; after the violent evolution of gas has ceased, the semi-solid product is shaken with dilute hydrochloric acid, and filtered; through the filtrate is passed a current of air, warming at the same time. A red precipitate of the imide is formed, which is washed with water and crystallized from hot glacial acetic acid.

A mere trace of the indicator is required, as its solubility in alcohol is very slight; the alkaline salts are readily soluble in this menstruum. H. D. R.

**Contributions to the Determination of Nitrogen and Albuminoids in Milk and its Products. L. Carcano.** (*Stazioni Sperimentali Agrarie Italiane*, xxii. 260.)—The author has compared the methods of Dumas and Kjeldahl for the estimation of nitrogen in milk and cheese, and that of Ritthausen for the determination of albuminoids in milk.

The average of his results are:—

| | | Ritthausen. °/₀ Albuminoids. | Dumas. °/₀ Nitrogen. | Kjeldahl. °/₀ Nitrogen. |
|---|---|---|---|---|
| Milk | 4 samples | 3·951 | ·6044 | ·5964. |
| Cheese | 10 samples | — | 4·927 | 4·901. |

H. D. R.

[Note by Abstractor.—The percentages of nitrogen in milk, multiplied by 6·38, the factor for casein and albumin given by Sebelein, give 3·856 and 3·805 °/₀ albuminoids respectively. As the whole of the nitrogen is not contained in the precipitate by Ritthausen's method, it follows that the latter gives too high results. The factor given by Sebelein is based on experiments of Hammarsten, and other Scandinavian chemists, and has lately been confirmed by Chittenden and his fellow-workers.]

**Is Saccharin Injurious?** (*From a Bill on Wine, before the German Parliament.*)—The views concerning the physiological action of saccharin are at present much divided, as the following abstracts from literature will show: According to Stutzer (*Saccharin, Leipzig*, 1885), and Aducco and Mosso (*Gaz. delle cliniche di Torino*, 1886, 14 and 15), saccharin is not hurtful. Millardet states that the action of pepsin upon albumin is but little affected by it, nor is that of pancreatin upon casein, whilst that of pancreatin upon starch is much impeded, a ten-fold amount of pancreatin being necessary to dissolve a given quantity of starch in the presence of saccharin, than without the latter (*V. der Nahrungs und Genussmittel*, 1888, *p.* 44). According to Girard, saccharin is not harmless after long continued use; it produces indigestion and is not excreted by the kidneys (p. 387), whilst Pavy is of opinion that saccharin is harmless and most valuable in diabetes (p. 387). Experiments by Plugge show that a 0·3 p.c. solution of saccharin entirely prevents the action of ptyalin, and retards that of pepsin and pancreatin (1889, 319). E. Gans, L. C. Wooldridge, Th. Stevenson, A. Petschek, and T. J. Zerner regard saccharin as harmless (458). F. Jessen states that "soluble" saccharin is without influence upon the action of ptyalin and but slightly retarding upon that of pepsin, and that the utilisation of food is not affected even by large doses. K. B. Lehmann declares it to be unobjectionable from a hygienic point of view, but its substitution for sugar should be allowed only after notice to the consumer (1890, p. 186 and 187.)

According to A. Stutzer, commercial saccharin contains about 40 per cent. of parasulphamidobenzoic acid, which disturbs digestion. Salkowski states the amount of the para-acid to be, in the older samples up to 60 per cent., in the newer ones 33 p.c.,

whilst "soluble" saccharin contains about 26 p. c. He could not trace any injurious effects upon the human organism (1887. p. 457.)

In addition to these opinions, there are a number of reports of medical and other societies, and of various governments, referring to the use of saccharin in articles of food. The Comite Consultatif d'hygiene publique de France reported on August 13th, 1888, that saccharin must not be regarded as a food, and that it could not be used as a substitute of sugar. Its use prevented or retarded the digestion of starchy and albuminous foods, and produced a profound disturbance of the organs of digestion and caused dyspepsia. It therefore recommended that the use of saccharin in articles of food should be prohibited (*Ver. d. K. Gesundheitsamts*, 1888, 608.)

The Royal Academy of Medicine of Madrid declared that the addition of saccharin to food should be regarded as an adulteration and that the importation of articles containing it should be prohibited (1889, 319.) The Hungarian Sanitary Council also recommended, in 1889 that the use of saccharin in food be prohibited, first, because it is not a sugar, but might be used by the public as such; second, because it had no food value; thirdly, because it was liable to produce disturbances of digestion on long-continued use; and lastly, on account of the probability that it would be used for fraudulent purposes (1889, 684). In the opinion of the Society of Bavarian Chemists (Meeting on May 16th and 17th, 1890), all articles of food, which owe their sweetness wholly or partially to saccharin, and which are not declared by the vendor to contain it, are to be regarded as adulterated (*Vahrungs and Genussmittel*, 1890, 187). The Austrian Sanitary Council considers saccharin to be harmless both to the healthy and to diabetic patients, but its use in food should be held to be an adulteration (*Gesundheitsamts*, 1890, 251).

The International Congress of Agriculture and Forestry, held in Vienna in September, 1890, passed a resolution, recommending that saccharin and similar sweetening agents should be allowed to be sold only by apothecaries upon physicians' prescriptions (1891, p. 118). According to a Spanish Cabinet order of April 3rd, 1889, saccharin is to be regarded as a drug, and articles of food may not contain the same, nor is their importation into Spain permitted (1889, p. 575). The Royal Academie of Medicine of Rio de Janeiro recommends the prohibition of the importation of saccharin into Brazil, as an article injurious to health (*Rev. intern. des falsif*, 1889, *p.* 146). By Royal Order of September 29th, 1889, the importation and production of saccharin was prohibited in Italy, and this order passed into law on May 15th, 1890 (*Ver. d. K. Gesundheitsamts*, 1890, *p.* 481). A further order was made in August, 1890, forbidding the use of saccharin in wine. In Russia the use of saccharin is prohibited, except for medicinal purposes (p. 723). Also Portugal prohibits the importation of saccharin. A Belgian regulation of December 10th, 1890, renders it incumbent upon the vendors of articles of food containing saccharin to declare the use of the same (*K. Gesund, A.*, 1891, *p.* 337).

The question has also occupied the attention of the German Government and experiments upon dogs have been made, but no injurious effect could be proved. On this

account the Government in question does not think that the use of saccharin should be absolutely prohibited in articles of food or drink, especially as for diabetic patients the article is doubtless a valuable one. But in order that the article be not used for purposes of fraud and deception in commerce, its use for sweetening wine is recommended to be prohibited, unless a declaration as to its presence be made. (*From the Zeitschr. f. angew. Chemie*, May, 1892.) O. H.

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**A Method of Discriminating between Natural and Artificial Water-marks. W. Herzberg.** (*Mitt. Königl. tech. Vesuchs.*, 1892, x., 45, through *Chem. Zeit.*).—The water-mark in papers is produced by the impression of the characters constituting it upon the surface of the paper. When this is done during the process of manufacture of the paper, the resulting water-marks may be termed "natural," while if it be caused by pressure on a finished paper it may be described as an "artificial" water-mark. The two kinds may be distinguished by immersing the paper in strong caustic soda solution, *e.g.*, 60 grms. of caustic soda and 200 c.c. of water. The "natural" water-mark becomes more noticeable by this treatment, while the "artificial" water-mark vanishes. The cause of this difference of behaviour is that the "natural" water-mark consists of a portion of the paper which is actually thinner than the surrounding parts, whereas in the case of the "artificial" water-mark the thickness of the paper was originally the same at that spot as at any other, and the action of the caustic soda swells the compressed fibre to its former dimensions. B. B.

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**The Quantitative Separation of Silver and Lead. R. Benedikt and L. Gans.** (*Chem. Zeit.* 1892, xvi. 181, 182.)—The method depends upon the fact that dilute nitric acid decomposes lead iodide, but does not attack silver iodide. Potassium iodide in excess is added to the solution containing both metals, and the resulting precipitate digested with dilute nitric acid, which must, of course, be free from chlorine. The presence of other metals of the same group, with the exception of mercury, does not interfere. Test analyses, made on known mixtures of the nitrates of lead, silver and copper, gave satisfactory results. The process, as applied to the determination of silver in argentiferous lead, is carried out as follows:—Ten to fifty grms. of the metal, according to the amount of silver it is supposed to contain, are dissolved in dilute nitric acid, containing tartaric acid to keep the antimony that may be present in solution, the solution diluted with boiling water, filtered and the filtrate diluted to 300 to 500 c.c., allowed to cool, then treated with 10 c.c. of a 10% solution of potassium iodide, warmed on the water-bath, and the residue of silver iodide collected and weighed in the usual manner. The percentage of silver in galena can also be determined by this method, the mineral being oxidised by nitric acid containing tartaric acid, and the solution treated with potassium iodide as before. A table is given in the original paper, showing the method to be fairly concordant with the dry assay for antimonial crude lead, ordinary crude lead, desilverized lead, and galena. B. B.

**The Composition of Coffee "Extracts." A. Domergue.** (*J. Ph. Chim.* 1892, xxv. 243, through *Chem. Zeit.*)—Coffee "extracts" or "essences" differ in composition according to their mode of preparation. One method consists in distilling the roasted and ground coffee with water, and thus obtaining a colourless distillate the odour of which recalls that of coffee, and which is turbid with a little essential oil. The residue in the retort is filtered and mixed with the distillate, thus forming an "extract." It does not contain all the extractive matter of the coffee, and when diluted with the appropriate amount of water is not the same colour as the freshly prepared liquid. In order to remedy this defect, caramel is added as well as strong alcohol, in order to preserve it. The author has examined six extracts, the results of which are given below. Samples A and B were prepared in the laboratory.

| | A. | B. | C. | D. | E. | F. |
|---|---|---|---|---|---|---|
| Extract dried at 100° C. | 13·7 | 17·6 | 41·01 | 27·2 | 30·1 | 19·26 |
| Ash .. .. .. | 0·61 | 0·79 | 4·3 | 3·1 | 1·4 | 1·83 |
| Caffeine .. .. | 0·106 | 0·105 | 0·060 | 0·040 | 0·05 | 0·096 |

The samples C, D, and E have been coloured with caramel.

Probably the best method of ascertaining the value of an extract consists in the determination of the caffeine, although it is to be noted that even samples A and D only contained about 1/10th of the amount of caffeine which is contained in an equal weight of coffee. No rule can be laid down as to what should constitute a normal coffee "extract."

B. B.

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**The Influence of Saccharine Matter in the Fodder upon the Composition of Milk Fat. A. Meyer.** (*Milch. Zeit.*, 1892, xxi. 49, through *Chem. Zeit.*)—The author is of opinion that the composition of milk fat is largely dependent upon the nature of the fodder, notably in the case of easily soluble carbo-hydrates, which tend to raise the proportion of liquid fatty acids, and thereby depress the melting point of the fat as a whole. The following are his conclusions in detail:—(1) A not unimportant quality in butter, as far as its commercial value is concerned, is its consistency. This is especially true in the English market, where soft butters appear to be less esteemed than those of normal consistency. (2) The consistency of butter, although influenced by the process of manufacture, mainly depends on the melting point of the butter fat. (3) The melting point of butter fat is obviously dependent on its composition, so that the presence of a considerable amount of volatile fatty acids, or of non-volatile liquid fatty acids, is disadvantageous. (4) The melting point of butter fat is affected both by the character of the individual animal yielding it and by the breed to which it belongs, and may be influenced by the selection of particular breeds and by the choice of individuals belonging to them. (5) The melting point of butter fat is also largely dependent upon the fodder, and easily digestible carbo-hydrates, especially saccharine substances, tend to lower the melting point of the butter, while fodder poor in soluble carbo-hydrates has the opposite effect.

B. B.

**The Simultaneous Precipitation of Copper and Antimony by the Electric Current. W. Hampe.** (*Chem. Zeit.*, 1892, xvi. 417-418.)—All commercial samples of copper, prepared electrolytically, which the author has examined, contain small quantities of antimony, ranging from 0·007 to 0·02 per cent. The reason for this is to be found in the practice of allowing the sulphuric acid, used as an electrolyte in the purification of argentiferous copper in the wet way, to circulate repeatedly through the depositing tank until it becomes comparatively rich in impurities. By frequent renewal of the liquor the co-precipitation of the antimony can be minimised, though it cannot be entirely avoided. The same action occurs in the electrolytic determination of copper from a solution containing antimony, even when the current is interrupted immediately precipitation is complete. Experiments made with copper solutions containing known amounts of antimony showed that under favourable circumstances as much as 0·02 per cent. might be precipitated.

B. B.

## LEGAL.

### HIGH COURT OF JUSTICE.*

*Tuesday, May* 31, 1892.

Queen's Bench Division. (Before the Lord Chief Justice)

Johnstone v. Tilden and others.

*Mr. Jelf, Q.C.. Mr. Pocock*, and *Dr. B. O'Connor* (instructed by Mr. J. P. Godfrey) appeared for the Plaintiff.

*The Solicitor-General* (*Sir Edward Clarke, Q.C.*) and *Mr. Boydell Houghton* (instructed by Messrs. Markby, Stewart, and Co.), appeared for the Defendants.

(*Transcript from Shorthand Writer's Notes*).

The Solicitor-General :—Will your Lordship forgive my interposing for the moment during the hearing of another case to relieve your Lordship's list of a case which we shall not have to trouble the jury with ? It is the case of Johnstone v. Tilden and others, and my learned friend Mr. Jelf, who appears for the plaintiff, wishes to say a few words.

The Lord Chief Justice :—Certainly.

Mr. Jelf :—If your Lordship thinks that I am not unduly interfering with the case that is now on, to suit the Solicitor-General and my convenience, I should like to mention this case now. I appear for the plaintiff, Dr. Johnstone, with my learned friends Mr. Pocock and Dr. O'Connor. It is an action brought by the Doctor against the Institute of Chemistry of Great Britain and Ireland, represented here by my learned friend the Solicitor-General. The action was brought to prevent the Institute from persevering in excluding him from being a Member and Fellow of the Institute upon a certain charge of unprofessional conduct which had been made against him. My Lord, I have very carefully

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considered the facts, and the evidence on which this case was sought to be based, and although my client was very desirous of clearing his character as much as he possibly could, I pointed out to him that their Charter gave the Institute very large powers, so long as they were acting in good faith and in accordance with their rules, in deciding rightly or wrongly on the matters brought before them. My client has, I hope wisely, placed himself in my hands in the matter, and in accordance with what has passed between the Solicitor-General and myself, we have agreed, with your Lordship's permission, to deal with the case by making a short statement on each side. On the part of the plaintiff, I desire to withdraw unreservedly any suggestion that these gentlemen have been acting otherwise than in good faith and in accordance with the rules of the Institute. On the other hand, I am glad to say that the Solicitor-General sees his way to saying this on behalf of the gentlemen whom he represents: "That their action was founded entirely on their view of the professional conduct of the plaintiff on a single occasion, and involved no imputation upon his character or professional capacity." Two views might possibly be taken of the question that was at issue; but looking to the circumstances of the case, and, it seems to me, the very handsome way I have been met by the Solicitor-General, with the consent of his clients, I think it would be extremely unwise for my client to fight for more than he has received. Under these circumstances we have agreed, with your Lordship's consent, to withdraw the record, and meanwhile the plaintiff undertakes to give up any title he might have had to be a Fellow or a Member of the Institute, and to hand over what perhaps I cannot say that my client is entitled to keep, but no doubt he will do what is right,—he will not persist in representing himself as a member of the Institute, and he will hand over his documents.

The Solicitor-General:—I am much obliged to your Lordship for allowing me to interpose. Representing the authorities of the Institute of Chemistry, my only desire is to make it perfectly clear, and it is their only wish to make it clear, that they acted throughout deliberately and in the exercise of the deliberate judgment vested in them, and according to the rules of the Institute to which the plaintiff himself belonged. The words my learned friend has read are words that I agree to; I agree that there might be some controversy and difference of opinion as to the action of the plaintiff himself, which was brought under the notice of the defendants, but it was carefully considered by them; they took the course they were bound to take in the interests of their profession, and I am glad to have the opportunity of saying not only that they have acted regularly in the manner, but they are anxious their action should not be taken to carry with it an imputation which it really ought not properly to bear. It was a question of professional conduct in a single instance and their action upon it, and that action they were obliged to notify. In this case, my Lord, we have agreed to the record being withdrawn with your Lordship's permission.

Mr. Jelf:—And no costs.

The Lord Chief Justice:—I can only give effect to the desire of the parties and the record may be withdrawn.

## CORRESPONDENCE.

BEECHFIELD,
WALTHAMSTOW,
*June* 18*th*, 1892.

*To the Editors of* THE ANALYST.

GENTLEMEN,

Dr. Frankland, in his letter in the June number of THE ANALYST, evades the true issue between us. My complaint was, that he stated my analyses to be incomplete, as they gave "no information regarding the quantities of organic elements in the samples," and further, that "failing *these data*, it is impossible to form any trustworthy opinion as to the quality of the water from a hygienic point of view." (*Vide* letter from Dr. Frankland, ANALYST, p. 90.)

It is perfectly plain that Dr. Frankland means by "organic elements" the "organic carbon and organic nitrogen," not the nitric nitrogen. His insinuation that my analyses consisted only of the data enumerated in his letter to you, instead of the details given in my paper, is neither correct nor to the point.

Those who were present when I laid the matter before the Society will remember that I placed upon the table copies of the printed report sent to Dr. Frankland, and they can bear me out when I repeat that the details were correctly described in my communication.

Dr. Frankland suggests that I am guilty of equivocation as regards the "nitrogen as nitrates," and implies that if I had estimated this "trustworthy datum" he would have been able to forgive the omission of the organic elements. It is true that I only tested qualitatively for nitric acid, but this datum had nothing whatever to do with the conclusion at which I was forced to arrive as to the inefficient purification of the samples of water in question. In common with almost every other analyst, I attach the greatest possible value to the determination of nitrates in samples of water, but, in the case of a water-supply from the river, the variation in the nitric nitrogen is so small that in any case it would not affect the conclusion drawn from the amount of free and albuminoid ammonia and of oxygen absorbed.

The statement in the last paragraph of Dr. Frankland's letter affects all chemists who analyse and report upon samples of water. If it were true, as there stated, that in cases of importance, as in the water-supply of towns, the only reliable method is the combustion process, it would be as well for the Water Committee to be re-constituted to inquire whether any changes have been made in the process in question which justify its being raised from the position to which it was relegated by that Committee in 1881.

I am, &c., yours,

LEO TAYLOR.